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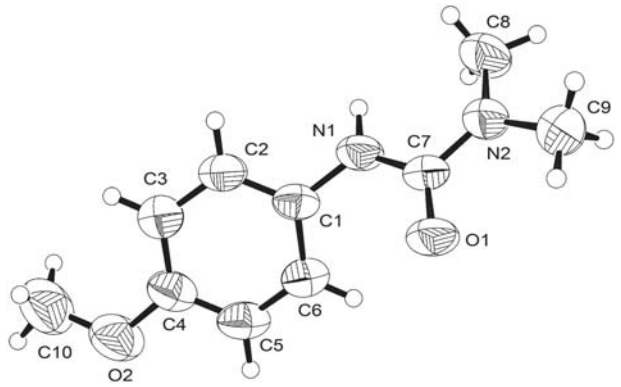
Crystal structure of 1,1-dimethyl-3-(4-methoxyphenyl)urea, $C_{10}H_{14}N_2O_2$



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Abstract

$C_{10}H_{14}N_2O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 14.9185(12)$ Å, $b = 7.7243(6)$ Å, $c = 9.2229(5)$ Å, $\beta = 91.032(6)°$, $V = 1062.63(13)$ Å^3, $Z = 4$, $T = 293(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.29 × 0.13 × 0.09 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	SuperNova, φ and ω
θ_{max}, completeness:	29.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5082, 2534, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1512
$N(param)_{refined}$:	135
Programs:	CrysAlis^{PRO} [17], SHELX [18], WinGX [19], CHEMDRAW Ultra [20].

Source of material

To a stirred solution of triphosgene (1.0 mole equivalent) in dichloromethane (DCM), a solution of 4-methoxyaniline (2.5 mole equivalents) and triethylamine (5.5 mole equivalents) in DCM was slowly added over 30 min at 0 °C. The mixture was stirred at 0 °C for 2 h, after which a solution of dimethylamine (3.0 mole equivalents) in tetrahydrofuran was added. The reaction mixture was stirred at 0 °C for 1 h. The mixture was poured onto water and the DCM layer was separated, washed with water, and dried over magnesium sulfate and the solvent was then removed under reduced pressure. Crystallization of the obtained raw solid using a mixture of ethyl acetate and diethyl ether (1:3 by volume) gave the title compound, 1,1-dimethyl-3-(4-methoxyphenyl)urea (83%) as colorless crystals, Mp. 132–133 °C (lit. 131–132 °C [1]).

Experimental details

Non-hydrogen atoms were refined with anisotropic displacement parameters. The N-H proton was refined freely but all other hydrogen atoms were placed in calculated positions and refined using a riding model. C-H bonds of the methyl groups were fixed at 0.96 Å, with displacement parameters 1.5 times U_{eq}(C) for the hydrogen atoms, and were allowed to spin about the C-C bond. Aromatic C-H distances were set to 0.93 Å and their U(iso) values of the corresponding hydrogen atoms were set to 1.2 times the U_{eq} values of the atoms to which they are bonded.

Comment

Many urea derivatives are very important intermediates in organic syntheses and show a variety of biological activities

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.24439(13)	0.3566(3)	0.26176(18)	0.0553(5)
C2	0.24067(15)	0.5161(3)	0.1958(2)	0.0665(6)
H2	0.2782	0.5391	0.1188	0.080*
C3	0.18216(16)	0.6430(3)	0.2417(2)	0.0710(6)
H3	0.1799	0.7496	0.1948	0.085*
C4	0.12752(14)	0.6108(3)	0.3569(2)	0.0670(6)
C5	0.13152(14)	0.4524(3)	0.4251(2)	0.0700(6)
H5	0.0953	0.4312	0.5042	0.084*
C6	0.18829(14)	0.3253(3)	0.3780(2)	0.0625(6)
H6	0.1893	0.2180	0.4237	0.075*
C7	0.35326(14)	0.1205(3)	0.29039(19)	0.0556(5)
C8	0.37929(19)	−0.0498(3)	0.0685(2)	0.0848(8)
H8A	0.3156	−0.0574	0.0529	0.127*
H8B	0.4063	−0.1596	0.0477	0.127*
H8C	0.4034	0.0370	0.0058	0.127*
C9	0.4621(2)	−0.1113(4)	0.2971(3)	0.1023(10)
H9A	0.4701	−0.0680	0.3940	0.153*
H9B	0.5185	−0.1095	0.2486	0.153*
H9C	0.4400	−0.2279	0.3005	0.153*
C10	0.0505(3)	0.8791(4)	0.3308(4)	0.1230(12)
H10A	0.1025	0.9520	0.3381	0.184*
H10B	−0.0002	0.9389	0.3695	0.184*
H10C	0.0387	0.8511	0.2309	0.184*
N1	0.30120(12)	0.2264(2)	0.20641(18)	0.0627(5)
N2	0.39827(13)	−0.0034(2)	0.21882(17)	0.0670(5)
O1	0.36005(11)	0.1371(2)	0.42292(13)	0.0739(5)
O2	0.06579(12)	0.7263(3)	0.40972(19)	0.0957(6)
H1	0.3151(16)	0.237(3)	0.117(3)	0.077(7)*
C1	0.24439(13)	0.3566(3)	0.26176(18)	0.0553(5)
C2	0.24067(15)	0.5161(3)	0.1958(2)	0.0665(6)
H2	0.2782	0.5391	0.1188	0.080*
C3	0.18216(16)	0.6430(3)	0.2417(2)	0.0710(6)
H3	0.1799	0.7496	0.1948	0.085*
C4	0.12752(14)	0.6108(3)	0.3569(2)	0.0670(6)
C5	0.13152(14)	0.4524(3)	0.4251(2)	0.0700(6)
H5	0.0953	0.4312	0.5042	0.084*

[2–5]. Various efficient procedures for the synthesis of aromatic ureas are known [6–11]. Aromatic ureas can be easily modified *via* treatment with lithium reagents followed by reactions with electrophiles, a methodology we have used in the last few years [12–16].

The asymmetric unit contains one molecule of $C_{10}H_{14}N_2O_2$. In the molecule, the methoxybenzene group is almost planar with a C3—C4—O2—C10 torsion angle of 7.5(4)°. The rest of the molecule is not planar with twist angles 46.90(9)° between the phenyl and the NC(O)N groups and 10.68(34)° between NC(O)N and N(Me$_2$) groups. In the crystal, N-H···O hydrogen bonding is observed with geometry: N1—H1···O1 = 158(2)°, N1···O1 = 2.966(2) Å. The hydrogen bonds construct chains of molecules along [001]. All bond lengths and angles are in the expected ranges.

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